

March 19, 1946.

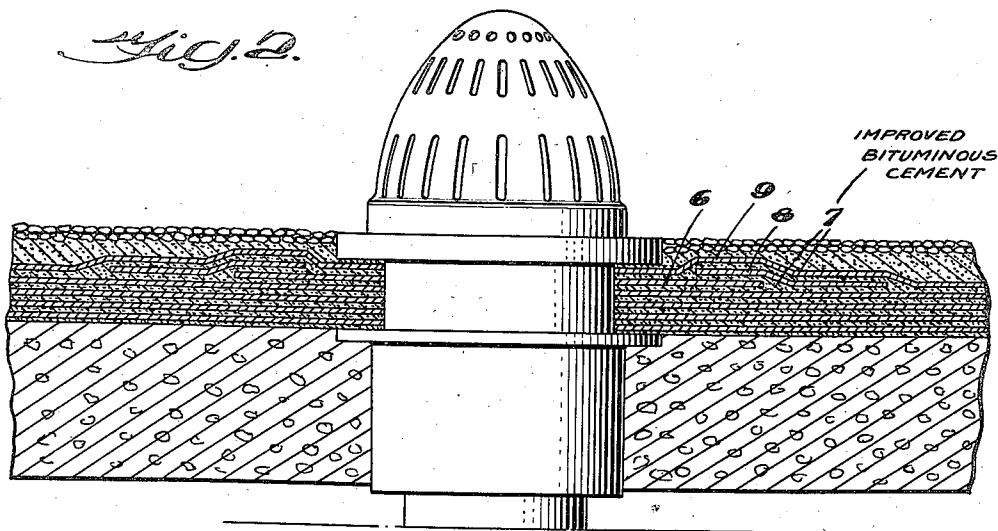
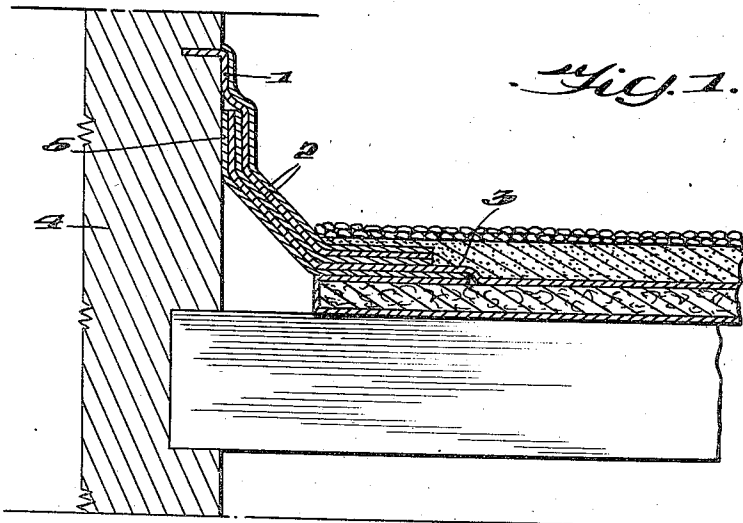
W. F. FAIR, JR

2,396,690

BITUMINOUS FLASHING CEMENT FOR COLD APPLICATION

Filed May 18, 1943

2 Sheets-Sheet 1



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2 Sheets-Sheet 2

Fig. 3.

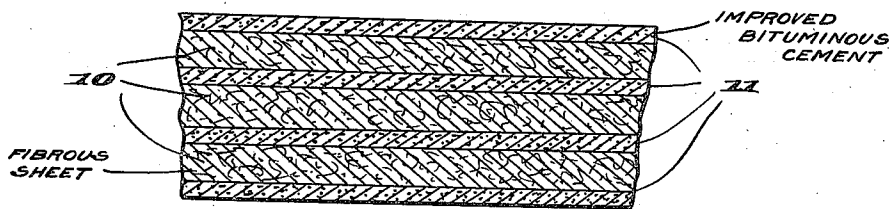


Fig. 4.

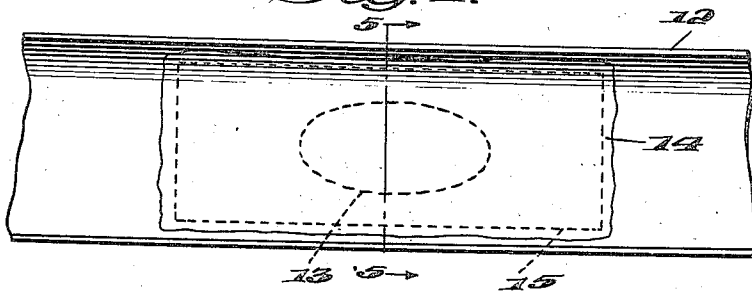


Fig. 6.

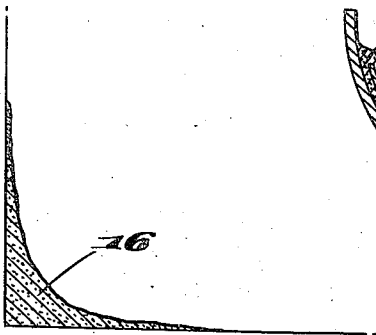
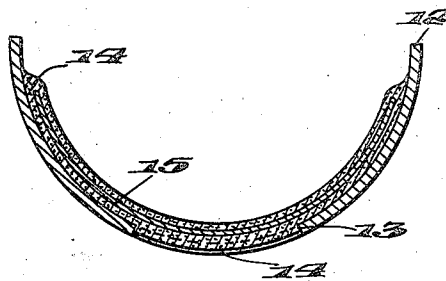


Fig. 5.



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UNITED STATES PATENT OFFICE

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BITUMINOUS FLASHING CEMENT FOR
COLD APPLICATION

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mesne assignments, to Koppers Company, Inc.,
a corporation of Delaware

Application May 18, 1943, Serial No. 487,530

4 Claims. (Cl. 106—280)

This invention relates to improvements in flashings, and bituminous cements or plastic mixtures particularly suitable for flashings, filleting and other structural means, and to improvements in manufacturing and applying flashings and bituminous cements.

The flashings of the present invention are non-metallic and serve to replace metallic flashings; or the herein described improved bituminous cements or plastic binders employed in making non-metallic flashings can be used in re-surfacing or repairing the latter or in repairing old corroded metal flashings, or gutters or for columns or cementing shingles. The cements are outstandingly suited for these purposes because, among other reasons, from a practical standpoint they can be uniformly applied simply by trowelling in summer or winter weather and no heating of the cement is required.

This cement or binder is prepared by compounding a specially prepared pitch with fibrous material and a relatively low-boiling solvent. The resulting mixture lends itself readily to adjustment to a wide range of temperature or other conditions by changing the penetration characteristics of the pitch, or by addition of mineral or other fillers, dusts or flours, and/or relatively high-boiling solvents or heavy oils, whenever necessary, and without destroying the bonding qualities of the cement. Also, the aforesaid relatively high-boiling solvents and relatively low-boiling solvents may be selectively employed to furnish cements of relatively rapid or relatively slow, initial setting properties while still retaining firm and lasting bonding and pliability characteristics of the cement. In other respects, it is superior to other previously manufactured bituminous cements, as for instance especially in non-flowing and nonembrittling characteristics. It is also highly flexible as well as tenacious.

The particular pitch employed in the cement for the purposes herein set forth is an improved so-called coal-digestion pitch. This pitch comprises coal dispersed by digestion in a heat-liquefiable bituminous medium which is preferably composed of, or contains, heavy hydrocarbon oil, or to which heavy hydrocarbon oil containing a preponderating proportion of aromatic hydrocarbons or constituents is added during or preferably after the digestion and dispersion process, or during or preferably after thermal decomposition of coal in such process.

In preparing the improved coal-digestion pitch, coal, such as bituminous coals or coking coals including either high or low volatile bituminous

coal, and certain commercial non-coking bituminous coals, is heated while admixed with tar or pitch. The temperature of the mixture while stirring the latter, is gradually increased over an extended period of time to substantially 300° C. or preferably to a temperature in the approximate range of 300° C. to 310° C. The temperature employed is generally not lower than about 270° C. nor higher than about 350° C. The tar or pitch used may be coal tar or pitch (such as a coke oven tar or pitch), heavy water gas tar or pitch, light water gas tar or pitch or blends of two or more of these tars and pitches. Other bitumens which may be used are, for example, oil-gas tar, Pintsch gas tar and wood tar, and corresponding pitches of these tars. Either before but preferably after dispersion of coal in the tar and/or pitch in the digestion process, is added the above-mentioned heavy hydrocarbon oil which is preferably heavy water gas tar heavy oil and which latter is preferred above light water gas tar heavy oil and over heavy coal tar oil.

The above-mentioned heavy hydrocarbon oils are high-boiling distillates obtained by distilling the corresponding tars and separating the distillate recoverable above approximately 300° C. Only a minor proportion (about 15 per cent or less) of the oil boils below 300° C. The boiling points may be within the approximate range of 250° C. to 450° C.

Heavy water gas tar heavy oils, which are substantially aromatic in character, are obtained by distillation from heavy water gas tar which in turn is obtained from water-gas generator plants in which Bunker-C or similar grades of residual petroleum fuel oil are used for carburetting. Heavy water gas tar is also known as residuum tar and is thus termed to distinguish it from what was formerly known as water gas tar or is known today specifically as light water gas tar. Light water gas tar heavy oil is obtained from light water gas tar which is produced in the carburetter of a water gas plant when petroleum distillates are used as carburetting agents.

Depending upon the proportions of coal, tar and/or pitch, and/or heavy oil, in the coal-digestion pitches, the latter pitch products have been prepared with Ring and Ball softening points in the approximate range of 35° C. to 150° C., all exhibiting improved rheological properties, as well as considerably better temperature susceptibilities (less change in viscosity with temperature change) and greater resistance to flow than the commonly used bitumens. In a cement having proper proportions of low-boiling solvent, of fi-

brous matter and of the improved coal-digestion pitch to render the cement capable of being trowelled, it is preferred that the softening point of the pitch binder be at least about 70° C. and that the penetration be at least about 10, preferably about 15, at 32° F., 200 grams, 60 seconds. The penetration at high temperatures should not be too high, for instance, not more than about 100, preferably not more than about 70, at 115° F., 50 grams, 5 seconds. Highly satisfactory results from the standpoint of practical workability and pliability after setting and cooling to low temperatures, for instance winter or freezing temperatures, are obtained with a cement containing a coal-digestion product prepared from a heavy water gas tar, heavy water gas tar heavy oil, and coal, which product has a softening point (Ring and Ball) of approximately 100° C., and penetrations of 15 at 32° F., 200 grams, 60 seconds; of 21 at 77° F., 100 grams, 5 seconds; and 40 at 115° F., 50 grams, 5 seconds. Excellent results are likewise obtained with a cement containing a coal-digestion pitch binder composed of similar constituents, and having a softening point of about 79° C. and penetrations of 10 at 32° F., 21 at 77° F., and 66 at 115° F.

A specific example of an improved coal-digestion pitch is one prepared by bringing together about 15,000 pounds of pulverized bituminous coal, about 4770 gallons of heavy water gas tar and about 500 gallons of heavy water gas tar heavy oil, and heating the mixture to a temperature in the range of approximately 300 to 310° C. within a period of eight to twelve hours. The heating in this range is continued for an additional two hours. Then the heating is discontinued whereupon approximately 1500 gallons of heavy water gas tar heavy oil totalling approximately 23% by weight are thoroughly mixed into the heated mixture. The product when cooled and solidified has a softening point of about 118° C., and penetrations at 32° F. of 19; at 77° F. of 30; and at 115° F. of 48.

Another example of an improved coal-digestion pitch is one prepared by bringing together 60 parts by weight of coke oven tar and 15 parts by weight of powdered bituminous coal and heating the mixture over a period of about three hours to a temperature of about 305° C. The heating is continued at about this temperature for an additional six hours. At the end of this period, about 14% by weight of heavy water gas tar heavy oil based on the weight of the heated material is added, distributed therein, and then the heating is discontinued. The resulting pitch has a softening point of about 74° C., and penetrations at 32° F. of 12; at 77° F. of 27; and at 115° F. of 72. If desired, the heavy water gas tar heavy oil may be substituted by light water gas tar heavy oil provided high melting coke oven pitch is included in the composition; though the heavy water gas tar heavy oil alone is preferred. Without the high melting coke oven pitch, less light water gas tar heavy oil may be included, which tends to result in a binder of poorer weather resistance.

An example of such an improved coal-digestion pitch is one prepared by charging a still with 120 parts by weight of crude light water gas tar and 124 parts approximately 28% by weight of light water gas tar heavy oil; adding to the mixture 120 parts by weight of high-melting coke oven pitch having a melting point of 145° C. (cube in air) and 80 parts by weight of pulverized coal; and heating this mixture to approximately 300°

C. in about one hour and maintaining the temperature at about 300° C. for approximately three hours with agitation, removing thereby about 13 parts of distillate. The residual product has a softening point of about 92° C. (Ring and Ball) and penetrations at 32° F. of 10, at 77° F. of 16, and at 115° F. of 31.

In some cases, one to two parts by weight of sulfur may be included in the digestion mixture, or air may be introduced for varying periods. The air and sulfur serve as dehydrogenating agents by which with subsequent addition of heavy water gas tar heavy oil, the characteristics of the pitch may alternatively be adjusted.

As an example of a flashing cement and its application, the following will serve for illustrative purposes: About 125 parts by weight of coal tar solvent naphtha are added to 290 parts by weight of a molten coal-digestion pitch such as any of those exemplified above. The solvent naphtha is uniformly distributed throughout the pitch by agitation, and then about 85 parts by weight of long fiber asbestos are added. The mixing is continued until the product is uniform throughout.

An example of a cement prepared with a coal-digestion pitch with a relatively low softening point, namely 55° C. (Ring and Ball) is one in which 17.5% by weight of asbestos fiber, 60.6% by weight of the pitch and 21.9% by weight of solvent naphtha are employed. The pitch is made of coal, heavy water gas tar, and heavy water gas tar heavy oil.

The solvent naphtha in the above examples is added in amount preferably to obtain a cement that can be trowelled. By adding larger amounts the cement is diluted as desired.

In preparing a flashing of a type shown in the accompanying diagrammatic drawings, any desired number of plies of fabric or felt or pressed fiber sheets, preferably impregnated with a water repellant bituminous saturant such as coal tars, water gas tars or pitches thereof, is used. A layer of the above described improved cement is trowelled on the surface of a sheet before another sheet is superimposed thereon. In Fig. 1 of the drawings, a built-in-place flashing is shown, in section, in place on a roof. This flashing comprises a ply 1 of fibrous material having on its under side and on its outer side a layer of the improved cement 2. A layer 3 of the roofing felt or tarred fabric is extended up the side of the parapet wall 4 under the outside ply. Initially, a layer 5 of the improved cement is placed between the parapet wall and the layer 3.

Fig. 2 illustrates more or less diagrammatically, the application of the improved flashing cement to drain installations. The drain installation shown is provided generally with an integrally mounted metal flange 6 that extends outwardly from the drain pipe and over a roof or deck. This metal flange is set in the improved cement 7. A fibrous layer 8 is placed over the edge of the flange 6 and is also set in the improved cement applied on top as well as underneath. A top covering layer 9 of roofing felt may then be applied.

Fig. 3 illustrates a laminated structure of a plurality of layers. Between the fibrous sheets or fibrous bitumen-treated layers 10 and on the outer surfaces of the outside sheets, the improved cement 11 is uniformly spread in relatively thin layers. The plies are built up in the cold, no heating being required. The outer surface of the cement slowly hardens and forms a seal. The

intermediate layers remain somewhat soft and definitely flexible. The product remains pliable and water resistant indefinitely, whether in ply or in mass construction, and the cement does not flow off in the hottest weather even immediately after application, or crack in the coldest weather, even after aging.

Figs. 4 and 5 illustrate the use of the improved cement as a repair means for metal gutters. Fig. 4 is a top view of a length of a gutter 12 showing a patch over a hole 13 in the bottom of the gutter. Fig. 5 shows a section of the gutter on line 5-5 of Fig. 4. The improved cement 14 is applied on both sides of a fabric or felt strip 15 large enough to extend well over the edges of the hole, and pressed into place. The edges of the strip 15 are also well covered with the cement. Several superposed plies may be so applied.

Tarred felt or woven fabric piles may be used for the fibrous membranes in any of the above structures.

Fig. 6 of the drawings, illustrates diagrammatically in section, the use of the improved cement 16 for protecting angles in building structures. The cement is trowelled into place, sets up moderately on the surface, and retains its original configuration for long periods through winter and summer weather without drying out, crumbling or breaking away, or becoming brittle.

The coal tar solvent naphtha in the cement may in whole or in part be replaced by other low-boiling coal tar solvents; or by equivalent or somewhat higher boiling fractions of heavy water gas tar, or light water gas tar, or petroleum distillates of high aromaticity or containing a preponderating proportion of aromatics. The boiling point ranges of these solvents preferably lie between about 100° C., and about 200° C., or from about 150° C. to about 300° C., depending upon the rate of set-up desired.

In the following table are given distillation data illustrating the above described solvents:

Distillation of typical solvents

Sample source	(1) Coal tar ° C.	(2) Coal tar ° C.	(3) Heavy W. G. tar ° C.	(4) Light W. G. tar ° C.
First drop	105	145	140	148
5%	127	160	153	175
10%	138	165	163	187
20%	148	169	173	201
30%	157	171	182	209
40%	164	174	191	218
50%	168	176	203	225
60%	173	177	207	228
70%	175	179	216	233
80%	179	182	225	241
90%	184	188	242	258
95%	195	198	258	280

If relatively quick drying products are desired, the lower boiling solvents of any of the sources mentioned may be selected for use, but if slower setting cements are required the higher boiling fractions are selected for incorporation in the cement. In the above table, solvents (1) and (2) provide a relatively quick drying cement; (3) provides a slightly slower drying cement; and (4) a considerably slower drying cement.

The mineral fiber, such as asbestos fiber, may be any of the grades known in the trade, but preferably a long fiber asbestos, such as a "5 R," is employed. The proportion of asbestos fiber may be as high as approximately 20% by weight of the cement and as low as approximately 5%. The proportion of the asbestos fiber in relation

to the coal-digestion pitch is such that in a residual layer remaining on a surface after evaporation of the low-boiling solvent the coal-digestion pitch at least completely coats the individual fibers and the latter uniformly distributed in the layer are in relatively close or at least in overlapping contact with each other. The asbestos may in some instances be partly replaced by a finely divided filler such as slate dust or clay or the like. A very smooth setting flashing cement that is easily trowelled is made by substituting the filler for about one-half of the asbestos in a given approved formula. The asbestos and additional heavy oil if incorporated in the cement as well as the solvent and filler may be varied in proportions to meet requirements for use and locality.

It has been found that seasonal changes in the formula can be readily made in the present improved cement simply by adjusting the penetration of the improved coal-digestion pitch binder selected or by changing the proportions of additional heavy oil and the amount of cut-back. A winter grade of cement capable of application at temperatures around 5° F. is made even from a pitch of low penetration characteristics by increasing the proportion of solvent naphtha and adding a small amount of heavy oil as in the formula—290 parts coal-digestion pitch (softening point 79° C., penetrations of 10 at 32° F., 21 at 77° F., and 66 at 115° F.), 10 parts about 3% by weight heavy water gas tar heavy oil, 150 parts solvent naphtha, 85 parts asbestos fiber. A summer grade cement is prepared from 290 parts coal-digestion pitch having the characteristics of the above last-mentioned pitch, 10 parts about 3% by weight heavy water gas tar heavy oil, 135 parts solvent naphtha, 85 parts asbestos fiber. As indicated above, instead of adding the small amount of heavy oil to either grade, a pitch of slightly higher penetration values may be used.

By choosing such a pitch having high penetration at 32° F., satisfactory low penetration at 115° F., and a relatively high softening point, a cement may be prepared, without incorporation of additional high boiling oils, which will be appropriate for year-round application, or for utilization even in the most varied climates. An example of a pitch of this type as given above has a softening point of 118° C., and penetrations of 19 at 32° F., 30 at 77° F., and 48 at 115° F.

If 56 parts of this pitch are incorporated with 28 parts of solvent and 16 parts of asbestos fibre as described above, a product will be obtained which can be practically applied over a range of atmospheric temperatures from below freezing to high summer roof temperatures, and moreover this cement after application, will not crack or become brittle at low temperatures because of its relatively high penetration at 32° F., and neither will it tend to flow or slump under the influence of hot summer temperatures because of its unusually relatively high softening point.

For use in close quarters or in confined spaces or areas, the use of a bituminous cement or plastic binder containing coal tar solvent naphtha is frequently objectionable from a health or convenience point of view, in which case a product made with an equivalent boiling heavy water gas tar light oil distillate containing no irritating coal tar acids or bases may be advantageously used. Such a product may be made as in the last example given immediately above by using

as the solvent the heavy water gas tar light oil described in the foregoing table as having a boiling range from 140° C. to 258° C.

The softening points and penetrations referred to above are determined in accordance with the standard tests of the American Society for Testing Materials.

What is claimed is:

1. A flashing cement for cold application having in admixture with asbestos fibre and a low-boiling hydrocarbon solvent vaporizable at atmospheric temperatures, a bituminous base consisting of coal-digestion pitch obtainable by heating a mixture of bituminous coal and a heat-liquefiable bituminous medium to a temperature in the range of about 300° C. to 310° C. and by distributing in said mixture about 14% by weight to about 23% by weight of heavy water gas tar heavy oil to form a homogeneous blend having in combination a softening point of at least 70° C. and penetrations at 32° F. of at least about 10 with 200 grams for 60 seconds, and at 115° F. of not more than about 70 with 50 grams for 5 seconds, the said solvent having a preponderating proportion of aromatic constituents and providing upon evaporation from the fiber-containing cement a residue that is adhesive, and pliable and that is non-flowing, non-brittle and flexible at atmospheric temperatures, the said solvent being present in sufficient proportion to render the cement readily spreadable on a surface, and the said coal-digestion pitch and asbestos fibers being in such proportions that on evaporation of the said solvent individual fibers in a residual layer of the cement are completely coated with coal-digestion pitch and are at least in overlapping contact with each other.

2. A flashing cement for cold application having in admixture with asbestos fiber and a low-boiling hydrocarbon solvent vaporizable at atmospheric temperatures a bituminous base consisting of coal-digestion pitch obtainable by heating a mixture of bituminous coal and a heat-liquefiable bituminous medium containing high-melting coal tar pitch and light water gas tar heavy oil to a temperature in the range of about 300° C. to 310° C. forming a homogeneous blend containing about 28% by weight of said heavy oil and having in combination a softening point of at least 70° C. and penetrations at 32° F. of at least about 10 with 200 grams for 60 seconds, and at 115° F. of not more than about 70 with 50 grams for 5 seconds, the said solvent having a preponderating proportion of aromatic constituents and providing upon evaporation from the fiber-containing cement a residue that is adhesive, and pliable and that is non-flowing, non-brittle and flexible at atmospheric temperatures, the said solvent being present in sufficient proportion to render the cement readily spreadable on a surface, and the said coal-digestion pitch and asbestos fibers being in such proportions that on evaporation of the said solvent individual fibers in a residual layer of the cement are com-

pletely coated with coal-digestion pitch and are at least in over lapping contact with each other.

3. A flashing cement for cold application having in admixture with asbestos fibre and a low-boiling hydrocarbon solvent vaporizable at atmospheric temperatures a bituminous base consisting of coal-digestion pitch obtainable by heating a mixture of bituminous coal and a heat-liquefiable bituminous medium to a temperature in the range of about 300° C. to 310° C. and by distributing in said mixture about 14% by weight to about 28% by weight of high-boiling hydrocarbon oil having a preponderating proportion of aromatics and selected from a group consisting of heavy water-gas tar heavy oil and light water-gas tar heavy oil, to form a homogeneous blend having in combination a softening point of at least 70° C. and penetrations at 32° F. of at least about 10 with 200 grams for 60 seconds, and at 115° F. of not more than about 70 with 50 grams for 5 seconds, the said solvent having a preponderating proportion of aromatic constituents and providing upon evaporation from the fiber-containing cement a residue that is adhesive, and pliable and that is non-flowing, non-brittle and flexible at atmospheric temperatures, the said solvent being present in sufficient proportion to render the cement readily spreadable on a surface, and the said coal-digestion pitch and asbestos fibers being in such proportions that on evaporation of the said solvent individual fibers in a residual layer of the cement are completely coated with coal-digestion pitch and are at least in overlapping contact with each other.

4. A bituminous flashing cement for cold application in which coal-digestion pitch, asbestos fiber, a low-boiling hydrocarbon solvent, and about 3% by weight of high-boiling oil selected from the group consisting of heavy water-gas tar heavy oil and light water-gas tar heavy oil, are homogeneously combined for trowelling on surfaces at atmospheric temperatures forming upon application and upon evaporation of said solvent at said temperatures a fiber-containing residue that is adhesive, and pliable and that is non-flowing, non-brittle and flexible at said temperatures, the said coal-digestion pitch, obtainable by heating a mixture of bituminous coal and a heat-liquefiable bituminous medium to a temperature in the range of about 300° C. to about 310° C. and by distributing in said mixture a high-boiling oil selected from the group consisting of heavy water-gas tar heavy oil and light water-gas tar heavy oil, having in combination a high softening point of at least about 70° C. and high penetrations at 32° F. with 200 grams for 60 seconds of at least about 10, and low penetrations at 115° F. with 50 grams for 5 seconds of not more than about 70, and said pitch containing initially about 14% to about 28% by weight of said high-boiling oil.

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